Distillation of Diesel Fuels

Scope

This method covers the distillation of diesel fuels and similar petroleum products.

Summary

A 100 ml sample is distilled under prescribed conditions which are appropriate to its nature. Systematic observations of temperature readings and volumes of condensate are made and from these data the results of the test are calculated and reported.

Comments

Samples of materials that visibly contain water are not suitable for testing. If the initial boiling point is above 150°F, shake the sample with anhydrous sodium sulfate or other suitable drying agent and separate it from the drying agent by decanting. It is possible to conduct this test on two samples at once using adjoining stills. Record all volumes in the graduate to the nearest 0.5 ml and all thermometer readings to the nearest 1.0°F.

Apparatus and Materials

A. Distillation flask: 125 ml.

B. 100 ml graduated cylinder.

C. Thermometer ASTM 8F.

D. Distillation apparatus suitable for petroleum products.

Procedure

A. Preheat the condenser bath on the distillation apparatus. The proper condenser bath temperature will depend on the wax content of the sample and on its distillation fractions. The minimum temperature which permits satisfactory operation should be used. In general, a bath temperature in the 32° to 40°F range

is suitable for kerosene and products meeting the specifications for Grade No. fuel oil and those meeting the specifications for Grade No. 1-D diesel fuel oil. In some cases involving Grade No. 2 fuel oil, Grade No. 2-D diesel fuel oil, etc., it may be necessary to hold the condenser bath temperature at some point in the 100° to 140°F range in order to avoid the condensation of solid waxy material in the condenser tube.

B. Remove any residual liquid in the condenser by swabbing with a piece of soft, lint-free cloth attached to a cord or copper wire.

C. Measure 100 ml of the sample in the graduated cylinder and transfer it as completely as practicable to the distillation flask, taking care that none of the liquid flows into the vapor tube.

D. Fit the thermometer, provided with a snug-fitting stopper, tightly into the neck of the flask so that the bulb is centered in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the vapor tube.

E. Place the flask in the distillation apparatus and, by means of a stopper through which the vapor tube has been passed, make a tight connection with the condenser tube. Make sure the flask is in a vertical position and that the vapor tube extends a distance of 1 to 2 inches into the condenser tube.

F. Place the graduate under the lower end of the condenser tube so that the end of the condenser tube is centered in the graduate and extends therein for a distance of at least 1 inch but not below the 100 ml mark. Cover the graduate with blotting paper, or similar material, suitably weighted, which has been cut to fit the condenser tube snugly.

G. Note and record the prevailing barometric pressure.

H. Turn the still heater on and start a stop watch. Apply heat to the flask and contents so that the time interval between the first application of heat and the initial boiling point is 5 - 15 minutes.

I. Immediately after the first drop comes over stop the stopwatch and record the time to initial boiling point and the boiling point temperature. Move the graduate so that the tip of the condenser touches its inner wall.

J. Continue to regulate the heating so that the rate of condensation into the graduate is uniform and averages 4 - 5 ml/minute. At the 5 ml condensate point start the

stopwatch and record the temperature. Record the volume of condensate as each minute is passed on the stop watch in order to keep track of the rate.

K. In the interval between the recovery of 5 ml and the end of the distillation observe and record the temperature at each multiple of 10% recovered from 10 to 90 inclusive. The stopwatch can be stopped at the 90 ml mark.

L. Start the stopwatch when the total residual liquid in the flask is about 5 ml and make a final adjustment in the heat, if necessary, so that the time from 5 ml of liquid in the flask to the end point is a maximum of 5 minutes. The end point is the maximum observed temperature after which the temperature begins to drop. Stop the stopwatch at the end point and record the temperature and the time from 5 ml remaining to the end point.

M. Turn off the heat, tilt the flask and allow it to cool.

N. While the condenser tube continues to drain into the graduate, observe the volume of condensate at 2 minute intervals until two successive observations agree. Measure this volume accurately, and record it, to the nearest 0.5 ml, as percent recovery.

O. After the flask has cooled, pour its contents into the condensate in the graduate and allow to drain until no appreciable increase in the volume of liquid in the graduate is observed. Record this volume, to the nearest 0.5 ml as percent total recovery.

Calculations

A. Deduct the percent total recovery from 100 to obtain the percent loss.

B. When the report is to be based on thermometer readings corrected to 760 mm Hg, obtain the correction to be applied to each thermometer reading by the following equation:

$$C_f = 0.00012\,(760 - P)(460 + t_f)$$

where:

C_f = Correction to be added algebraically to the observed thermometer reading t_f.

P = Prevailing barometric pressure, mm Hg, at the time of the test.

C. If the thermometer readings are corrected to 760 mm Hg pressure, the actual loss shall be corrected to 760 mm-Hg pressure according to the equation:

$$\text{Corrected loss} = AL + B$$

where:

L = Percent loss as calculated from test data and

A and B = Numerical constants, the values of which depend upon the prevailing barometric pressure. Consult the reference for a table of these values.

D. To report thermometer readings at prescribed percentages evaporated, calculate each required thermometer reading as follows:

$$T = TL + \frac{(TH-TL)(R-RL)}{(RH-RL)}$$

T = Thermometer reading at the prescribed percent evaporated.

R = Percent recovered corresponding to the prescribed percent evaporated.

RH = Percent recovered adjacent to, and higher than R, at which a thermometer reading "TH" was noted.

RL = Percent recovered adjacent to, and lower than R, at which a thermometer reading "TL" was noted.

E. All of the above calculations can be done by computer.

Bibliography

Annual Book of ASTM Standards (1987) Vol. 05.01, Sec. 5, ASTM, Philadelphia, PA, D 86